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Effect of annealing temperature on the size and magnetic properties of $CoFe_2O_4$ nanoparticle

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Abstract. $CoFe_2O_4$ (CFO) nanoparticles (NPs) are synthesized using sol gel method and are annealed at 400, 600 and 800 ^oC for 4h. The crystal structure and morphology of the NPs are investigated through XRD and TEM analysis. The X-ray diffraction analysis shows that all the samples are well formed and attain a cubic structure with Fd-3m space group. The morphology of the material is found to be polygonal and the particle size of the NPs is increased with increase of annealing temperature as 400, 600 and 800 to be 20 nm, 30 nm and 70 nm respectively. The magnetic properties of the NPs are investigated using VSM and observed that the curie temperature for 400, 600 and 800 ^OC annealing temperature are 762 K, 780 K, 769 K respectively. The Ms of 600 sample is 80 emu/g. The 400 and 800 sample shows lower Ms value this is due to poor crystalanity and exaggerated grain growth at the respective temperatures. The coercivity of the sample shows linear dependence with particle size of the material the highest coercivity is obtained for 400 sample and low value for 800 sample.

INTRODUCTION

Spinel ferrite nanoparticles (NPs) with the general formula MFe_2O_4 (M=Cr,Mn,Co,Ni,Cu and Zn) are one of the important class of materials because of their unique magnetic and electrical transport properties with a good chemical and thermal stabilities. Technologically, these materials are very promising and have been used in several applications, including magnetic recording, electronics, biomedicine, catalysis, sensors and pigments etc. [1–5]. $CoFe_2O_4$ NPs have good magnetostrictive, magneto crystalline anisotropy, high coercivity (>5 K Oe) and moderate saturation magnetization (80 emu/gm) among all the ferrite family, which makes it competent to create a new turn in the existing world of ferrites. Moreover, they exhibit excellent chemical stability and mechanical hardness; a large magneto-optical effect; a high curie temperature; and high electromagnetic performance [6,7]. Magnetic properties of nanocrystalline cobalt ferrite are sensitive to the crystal structure, many researchers have carried out the investigation on nanocrystalline $CoFe_2O_4$. Abbas et al. [8] have prepared nanocrystalline $CoFe_2O_4$ by co-precipitation and ceramic method. They have found that structural parameters depend upon synthesis method. Thing et al. [9] have prepared fine particle of cobalt ferrite by complex metric method using ethylene demine tetra acetic acid as a completing agent and found that $CoFe_2O_4$ powder claimed at above 850°C shows single phase crystallographic structure. Zip et al. [10] synthesised $CoFe_2O_4$ by a modified chemical co-precipitation route and found that thermal heating changed the cation distribution in tetrahedral to octahedral sites. Wage et al. [11] have synthesised cobalt ferrite NPs using mechanical alloying method and found that crystallite size depends on the milling time, ball-to-powder ratio, and annealing temperature. Very limited efforts have been made to compare the particle size as well as magnetic properties associated with annealing temperature. Hence the present paper discusses the variation of particle size and magnetic properties associated with different annealing temperature.

EXPERIMENT

In case of sol gel synthesis, stochiometric amount of high purity powders of cobalt nitrate ($Co(NO_3)_2.6H_2O$) (Alfa Aesar) and iron nitrate ($Fe(NO_3)_2.9H_2O$) (Alfa Aesar) were dissolved in a minimum amount of deionised water or ethylene glycol and stirred for half an hour. Then the solution was heated to about 60 ^OC and citric acid was added followed by ethylene glycol. A particular molar ratio of citric acid to total moles of nitrate ions was maintained. The temperature of the solution was then

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increased gradually to 120 ^{O}C and kept constant for a long time to get the xerogel. It was then dried and a black precipitate was obtained. The dried precursor was then annealed in a tube-furnace at 400,600 and 800 ^{O}C for 4 hours, here onwards the 400,600 and 800 annealed sample is denoted as CFO 400, CFO 600 and CFO 800. The crystal structure was studied by the X-ray diffraction (XRD) technique using Ni filtered CuKα radiation (PANalytical X' Pert PRO Diffractometer, Netherlands).The particle morphology and sizes of samples prepared were directly investigated by transmission electron microscopy (TEM, JEOL-2010). The magnetic characterizations were performed in the temperature range 2 K to 900 K using Vibrating Sample Magnetometer attached with physical property measurement system (Quantum Design, USA).

RESULTS AND DISCUSSION

Fig.1 shows the powder XRD patterns of $CoFe_2O_4$ NPs annealed at 400, 600 and 800^{O}C for 4h. The peaks are well matched with the standard JCPDS file (No.22-1086). The material crystallizes in cubic structure with Fd-3m space group. It is evident from Fig.1 that as annealing temperature increases the material become more and more crystalline and the peak broadening decreases and particle size increases. The 600 sample seems to be highly crystalline and the intensity of them is very high but in the case of $CoFe_2O_4$ 400 the peak is very broad and intensity is very less this may be due to the poor crystalanity and in the case of 800 sample the intensity is very less this may be due to the fact that at this temperature the compound starts to melt. Fig. 1(b) shows the FTIR image of 600 sample as a representative of the series. It is clear from the Fig. that all the peaks suggest the phase pure formation of $CoFe_2O_4$

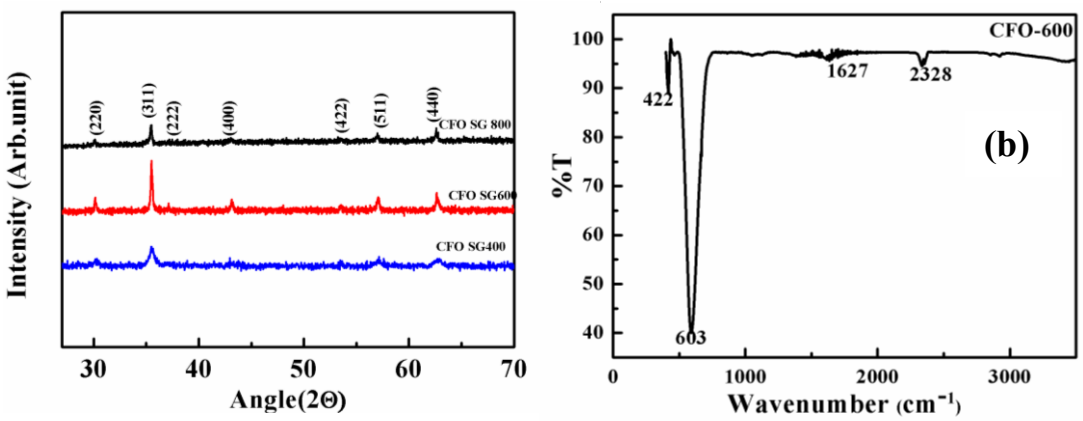

FIGURE 1 The X-ray diffraction pattern of CFO 400, CFO 600 and CFO 800 sample and FTIR image of SG 600 sample.

TEM images of CFO 400 and CFO 600 for 4h is shown in Fig.2 (a,b,). It is observed from the image that the morphology of the material is polygonal in type. Some moderately agglomerated particles as well as separated particles are present in the images. Agglomeration is understood to increase linearly with annealing temperature and hence some degree of agglomeration at 600 and 800 ^{o}C appears unavoidable. However, From Fig. it is noteworthy that the CFO 600 sample is more crystalline and the crystallite size is found to be 0.99 nm. The indexed SAED pattern (Fig.2(b,e)) is well matched with the powder XRD pattern shown in Fig.1. The particle size distribution of them is shown in Fig.2(g,h,i), It is observed that as particle size of the material increases with increase of annealing temperature this is because of grain growth happening at an elevated temperatures. The average particle size of CFO 400 is 20 nm, CFO 600 is 30 nm and CFO 800 is 70 nm. The CFO 800 data is not given in this manuscript it is already communicated.

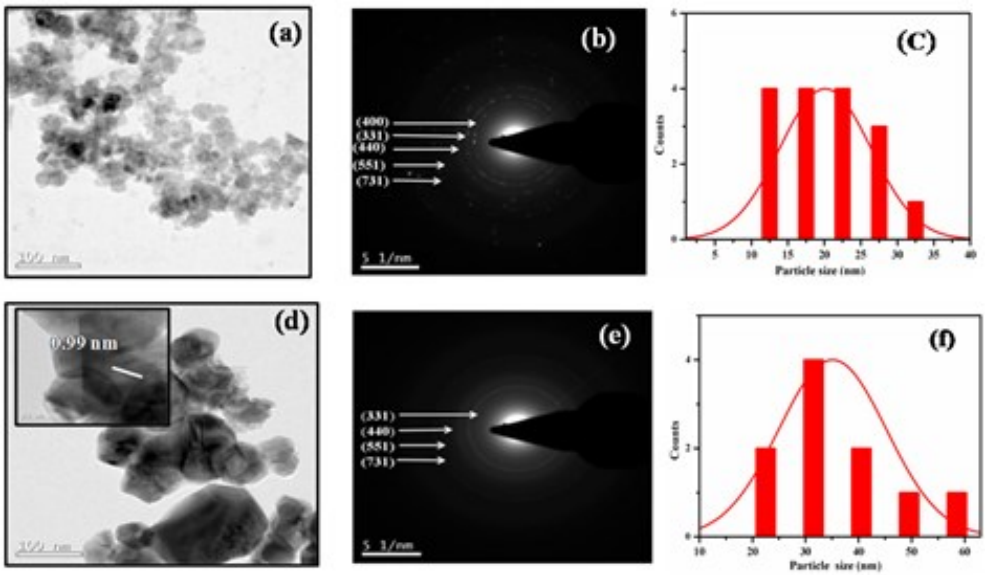

FIGURE 2 TEM images SAED pattern and particle size histogram of CFO 400 and CFO 600 sample.

The temperature dependent magnetization M(T), at 50 Oe is shown in Fig.3. for all the samples All the three samples show high temperature Tc. The CFO 400 has Tc of 762K, CFO 600 is 783 K and CFO 800 is 769 K respectively. The small difference in the Curie temperature of the samples is due to the variation in particle size caused by the different annealing condition which is clear from the TEM image. The FC magnetization of CFO 400 sample is 8 emu/g and in the case of CFO 600 and CFO 800 the FC magnetization is around 12 emu/g. This is because of the increased crystalanity of the sample at high temperatures.

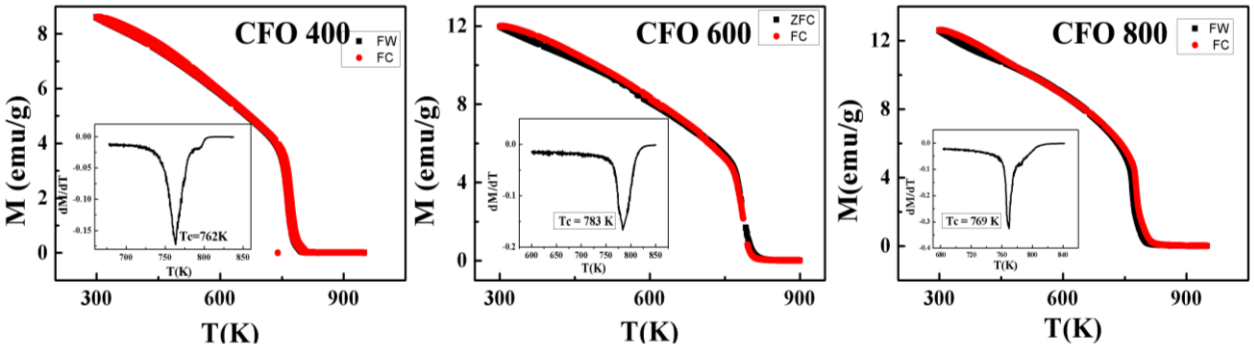

FIGURE 3. Temperature dependent magnetization M(T) of CFO 400, CFO 600 and CFO 800 sample.

Field dependent magnetization (M(H)) of CFO 400, CFO 600 and CFO 800 is shown in Fig.4. From Fig. it is observed that none of the sample shows superaramagnetic behavior, the saturation magnetization (Ms) for CFO 400 is 40 emu/g, while in CFO 600 is 80. The Ms for CFO 800 is 60 which is not given in Fig. in SG 400 the Ms value is very low because of the poor crystalanity of the material while at CFO 600 the sample shows maximum crystalanity and grain growth (Fig.1 and 2) therefore sample shows maximum Ms and finally in the case of CFO 800 the exaggerated grain growth taken place due to the increased annealing temperature of the sample and which degrade the Ms value of the material. The variation of coercivity is also shown in Fig. and it is clear from the Fig. that the coercivity value is depending on the particle size of the material. Coercivity value is high for CFO 400 and it is decreasing with increase of annealing temperature.

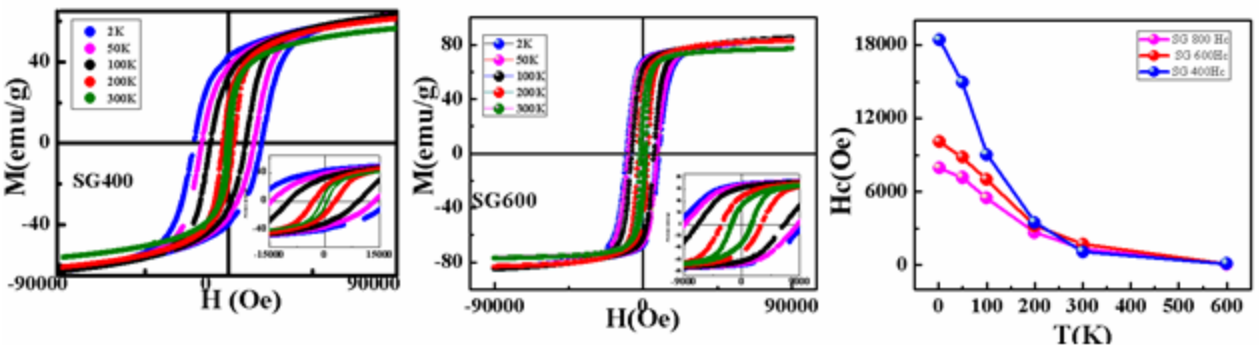

FIGURE 4. Field dependent magnetization of CFO 400, CFO 600 and CFO 800 sample

CONCLUSION

We report a comparative study of the CFO NPs annealed at 400, 600 and 800 ºC for 4h. X-ray diffraction analysis shows that all the samples are well formed and attain a cubic structure with Fd-3m space group. The morphology of the material is found to be polygon and the size of the material is increased with increase of annealing temperature, in CFO 400 particle size is 20 nm, for CFO 600 is 30 nm and CFO 800 is 70 nm. The magnetic properties of the material is investigated and the Tc for CFO 400, CFO 600 and CFO 800 is found to be 762 K,783,769K respectively. The Ms value of CFO 600 sample is 80 emu/g. The CFO 400 and CFO 800 sample shows low Ms value due to poor crystalanity and exaggerated grain growth happening at these temperatures. The coercivity of the sample shows linear dependence with particle size of the material.

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